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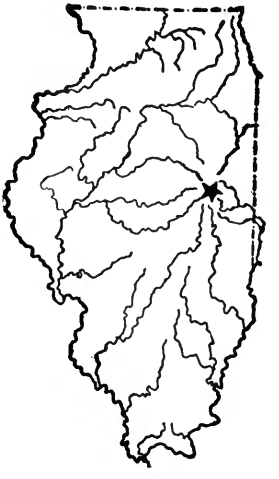

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A MODIFICATION OF THE BABCOCK TEST FOR THE DETERMINATION OF FAT IN BUTTERMILK

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An accurate method for the determination of fat in buttermilk is necessary in order to control the exhaustiveness of the churning process. The testing equipment in creameries usually consists of a Babcock test outfit, which affords a cheap and rapid method for the determination of fat in milk products. Certain procedures have been developed and certain types of test bottles have been produced for testing whole milk and cream, but for buttermilk the present Babcock method has not proved satisfactory, nor is there an adequate test bottle. Buttermilk is tested in the ordinary 18-gram skim-milk bottle, which is graduated from 0.0 to 0.25 of one percent (a few bottles graduated from 0.0 to 0.50 of one percent are being put on the market), whereas the amount of fat in buttermilk will, in some cases, exceed one percent.

This bulletin describes an attempt to devise a modification of the Babcock method that will prove more satisfactory than the present method. The experiment was so conducted as to obtain information on the following points:

A. The conditions limiting the use of the Babcock test for buttermilk.

1. The amount of sulfuric acid used.
2. The time of centrifuging.
3. The speed of centrifuging.

B. A comparison of the Babcock, normal butyl alcohol,[1] and Roese-Gottlieb methods for determining the amount of fat in buttermilk.

METHOD OF PROCEDURE

The buttermilk used in the experimental work was obtained from the churning of neutralized cream in a Dual churn and worker.[2]

All the Babcock and normal butyl alcohol tests were conducted in an electric centrifuge having a 22-inch disc and a speed adjustment ranging from 850 to 1700 revolutions per minute.

[1]Chicago Dairy Produce, December 31, 1921.

[2]With the exception of sample No. 12, Table 4, which was hand-churned buttermilk.

The acid was measured directly into the test bottle from a burette, as it was found that accurate measurements could not be obtained by the use of an acid measure or graduated cylinder. The sulfuric acid used had a specific gravity of 1.83.

Skim-milk bottles graduated from 0.0 to 0.50 of one percent were used for the normal butyl alcohol tests, and bottles graduated to 0.25 of one percent were used for the Babcock tests.

ROESE-GOTTLIEB DETERMINATIONS

In general, the procedure as given by the Association of Official Agricultural Chemists[1] was followed in the Roese-Gottlieb determinations. Three extractions with ether were made in each case. It was found necessary to use more ammonia than is needed in testing other milk products, and it was often necessary to add ammonia in the second and third extractions in order to break the emulsion which formed upon the addition of ether. The total amount of ammonia used for a 5-gram sample varied from 2 to 6 cc.

THE NORMAL BUTYL ALCOHOL TEST

The normal butyl alcohol procedure was that described by Professor W. J. Mitchell,[2] which modifies the Babcock method by using 9 cc. of buttermilk, 2 cc. of normal butyl alcohol, 7 to 9 cc. of sulfuric acid, and 6-2-2 minute whirling periods.

THE BABCOCK TEST

The common Babcock method of testing buttermilk makes use of an 18-gram sample, about 20 cc. of sulfuric acid, and a 10-10-5 minute whirling period. The unsatisfactory results of several preliminary tests following this method led to a study of the following variations of the test:

1. *Amount of Acid Used.*—Comparative tests were made, using 11 and 13.5 cc. of sulfuric acid with a 10-gram sample of buttermilk. (Since the graduated portion of the neck of an 18-gram skim-milk bottle is not of sufficient size to measure the fat in some samples of buttermilk, it was decided to use a 10-gram sample in an 18-gram bottle.)

2. *The Time of Centrifuging.*—A comparison was made of the following three periods of centrifuging: (a) 20–20–10 minutes, (b) 10–10–5 minutes, (c) 5–3–3 minutes.

3. *Speed of the Centrifuge.*—The effect of a variation in the speed of centrifuging was determined by a comparison of the results obtained at the following speeds: 850, 1150, 1700 R. P. M.

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[1]Methods of Analysis, A. O. A. C., 1920.
[2]Chicago Dairy Produce, December 31, 1921.

EXPERIMENTAL DATA

A. CONDITIONS LIMITING THE USE OF THE BABCOCK TEST FOR BUTTERMILK

1. *The Amount of Acid Used.*—As the maximum amount of acid that could be used without danger of charring the fat globules had been found to be 13.5 to 14 cc. (specific gravity 1.83), 13.5 cc. were selected as the maximum and 11 cc. were arbitrarily chosen as the minimum amount of acid to be used in the investigation. The influence of the quantity of acid used is shown by the following table.

TABLE 1.—INFLUENCE ON FAT TEST OF AMOUNT OF ACID USED
Speed of Centrifuge: 1150 R.P.M. 10-10-5 Minute Whirling Periods

Sample No.[1]	11 cc. acid	Average	13.5 cc. acid	Average
	%	%	%	%
1.....	.11		.36	
	.14	.125	.36	.360
2.....	.19		.33	
	.19	.190	.36	.345
3.....	.21		.47	
	.19	.200	.49	.480
4.....	.23		.43	
	.16	.195	.43	.430
5.....	.16		.47	
	.16	.160	.49	.480
6.....	.13		.40	
	.13	.130	.40	.400
7.....	.41		.54	
	.41	.410	.54	.540
Average...	...	.202	...	.434

[1]Sample numbers that recur in other tables refer to the same buttermilk.

Table 1 gives a comparison of the results obtained from running seven different samples at 1150 R.P.M., using the two different amounts of acid. It will be seen that a much lower reading was obtained from the use of 11 cc. of acid than when 13.5 cc. were used. The average percentage of fat obtained with the use of 13.5 cc. of acid was more than twice the average test obtained with 11 cc. of acid. These data clearly show the importance of using the maximum amount of acid.

2. *The Time of Centrifuging.*—The following table presents a comparison of the average tests obtained with three different periods of centrifuging at a speed of 850 R.P.M. using 13.5 cc. of acid, and of the average tests obtained from two different whirling periods at a speed of 1150 R.P.M. using both 11 and 13.5 cc. of acid.

These data show that the length of the period of centrifuging is of minor importance. Under the conditions indicated in Part I of the table, the longer whirling time resulted in slightly higher average

TABLE 2.—THE EFFECT OF VARYING THE LENGTH OF THE WHIRLING PERIOD[1]

Sample No.	Whirling period (minutes)			Speed of centrifuge	Acid used
	5–3–3	10–10–5	20–20–10		
Part I					
	%	%	%	R. P. M.	cc.
4	.380	.440	.410	850	13.5
6	.405	.405	.415	"	"
7	.500	.530	.560	"	"
8	.695	.750	.730	"	"
9	.380	.370	.450	"	"
10	.365	.355	.420	"	"
Average	.454	.475	.497	"	"
Part II					
		%	%	R. P. M.	cc.
1	....	.360	.335	1150	13.5
2	....	.345	.345	"	"
3	....	.480	.470	"	"
4	....	.430	.430	"	"
5	....	.480	.480	"	"
7	....	.530	.540	"	"
Average	....	.438	.433	"	"
Part III					
		%	%	R. P. M.	cc.
1	....	.125	.170	1150	11
2	....	.190	.205	"	"
3	....	.200	.290	"	"
4	....	.195	.255	"	"
5	....	.160	.290	"	"
6	....	.130	.130	"	"
7	....	.410	.495	"	"
Average	....	.201	.262	"	"

[1]The values given in this table are the averages of duplicate tests. The greatest difference between duplicates was 0.07 percent.

fat percentages. By comparing the averages given in Parts I and II it will be seen that the longer whirling periods gave higher results only at the lower speed and even then the increase was slight. The only really appreciable increase in the test obtained from a longer whirling period is shown by the figures given in Part III; these show that when the minimum amount of acid (11 cc.) was used the longer whirling period resulted in an increase in the average reading from 0.201 to 0.262 percent, an increase of about one-third. However, by referring to Part II of the table, it will be seen that when 13.5 cc. of acid were used at a speed of 1150 R.P.M. as high results were obtained from 10-10-5 minute periods of centrifuging as from 20-20-10 minute periods.

3. *The Speed of Centrifuging.*—To determine whether or not increasing the speed of centrifuging would increase the reading, the same samples were run at 850 R.P.M. and 1150 R.P.M. with 10-10-5 minute whirling periods, and at 1150 and 1700 R.P.M. with 20-20-10 minute whirling periods. In all cases 13.5 cc. of sulfuric acid (the amount found to give best results) were used. It was found that

when the speed of the centrifuge was increased to 1700 R.P.M. several of the test bottles were broken by the increased pressure exerted upon them. For this reason and because it was thought that any increase in reading to be obtained from the higher speed would be more noticeable with 20-20-10 minute whirling periods than with the shorter ones, it was deemed advisable to use the high speed only with the longer whirling period.

TABLE 3.—THE EFFECT OF VARYING THE SPEED OF CENTRIFUGING[1]

Sample No.	Speed of centrifuge			Whirling period	Amount of acid used
	850 R. P. M.	1150 R. P. M.	1700 R. P. M.		
	%	%	%	*minutes*	
4	.440	.430	....	10–10–5	13.5 cc. in all cases
6	.425	.400	....	"	
7	.530	.540	....	"	
8	.750	.690	....	"	
9	.370	.390	....	"	
10	.355	.350	....	"	
Average.	.478	.467	....	"	
1	....	.360	.355	20–20–10	13.5 cc. in all cases
2	....	.345	.325	"	
3	....	.470	.450	"	
4	....	.430	.435	"	
5	....	.480	.490	"	
7	....	.540	.555	"	
Average.	....	.437	.435	"	

[1]The values given in this table are the averages of duplicate tests. The greatest difference between duplicates was 0.05 percent.

The data in Table 3 show that when using 13.5 cc. of acid and a 10-10-5 minute whirling period as high results were obtained with a speed of 850 R.P.M. as with 1150 R.P.M. Increasing the speed to 1700 R.P.M. resulted in no higher readings than those obtained at 1150 R.P.M., even with the 20-20-10 minute whirling periods.

B. A COMPARISON OF THE BABCOCK, NORMAL BUTYL ALCOHOL, AND ROESE GOTTLIEB METHODS FOR DETERMINING THE PERCENTAGE OF FAT IN BUTTERMILK

As a result of the foregoing data, the following modification of the Babcock method was decided upon for a comparison with the normal butyl alcohol and Roese-Gottlieb methods of determining the fat in buttermilk: 10-gram sample; 13.5 cc. of acid (measured from a burette); 10-10-5 minute whirling periods; 1150 R. P. M. The results are given in Table 4.

A comparison of the results obtained by the Babcock and Roese-Gottlieb methods shows that the Babcock results are always less than those of the Roese-Gottlieb. It was noticed also that the difference between the two was fairly constant, being about 0.2 of one percent of fat. (The average difference between the results obtained on the 12 samples is 0.2047 of one percent). In other words, practically

0.2 of one percent of the fat in buttermilk is held so tightly by the milk constituents that the combined action of the acid and the centrifugal force fails to liberate it. The same fact is true in testing whole milk, in which test a correction is made for the approximate 0.2 of one percent fat that does not rise in the neck of the bottle, by reading the fat column to the top of the meniscus.

By using 0.2 as a correction factor and adding to the results obtained by the Babcock method, a value is obtained which is closely comparable to that obtained by the Roese-Gottlieb method.

In addition, the data in Table 4 confirm the work of Professor Mitchell, already referred to, in that the results obtained by the normal butyl alcohol method correspond closely with those obtained by chemical analysis.

TABLE 4.—A COMPARISON OF THE MODIFIED BABCOCK, NORMAL BUTYL ALCOHOL, AND ROESE-GOTTLIEB METHODS FOR DETERMINING THE FAT IN BUTTERMILK

Sample No.	Babcock	Average	Babcock plus .2	N. B. alcohol	Average	Roese-Gottlieb	Average
	%	%	%	%	%	%	%
1.....	.36	....	....	.58	....	.590	.....
	.36	.360	.56	.58	.580	.572	.5810
2.....	.36	....	....	.53	....	.545	.....
	.33	.345	.545	.53	.530	.544	.5445
3.....	.49	....	....	.69	....	.667	.....
	.47	.480	.68	.68	.685	.664	.6655
4.....	.43	....	....	.60	....	.628	.....
	.43	.430	.63	.60	.600	.626	.6270
5.....	.49	....	....	.68	....	.698	.....
	.47	.480	.68	.68	.680	.685	.6915
6.....	.40	....	....	.54	....	.596	.....
	.40	.400	.60	.54	.540	.598	.5970
7.....	.54	....	....	.72	....	.759	.....
	.54	.540	.74	.72	.720	.772	.7655
8.....	.71	....	....	.94	....	.904	.....
	.67	.690	.89	.94	.940	.897	.9005
9.....	.39	....	....	.56	....	.570	.....
	.39	.390	.59	.55	.555	.570	.5700
10.....	.35	....	....	.56	....	.566	.....
	.35	.350	.55	.56	.560	.554	.5600
11.....	.44	....	....	.62	....	.662	.....
	.44	.440	.64	.62	.620	.645	.6535
12.....	1.29	....	....	1.50	....	1.485	.....
	1.29	1.29	1.49	1.50	1.50	1.500	1.4925
Average.	....	.516	.716	....	.709	....	.7207

DISCUSSION OF THE TESTS

The results of this work indicate that the amount of acid added is the most important factor to be considered in using the Babcock test for buttermilk. Another very important factor, which does not appear in the data presented, is the construction of the skim-milk bottle used. Many bottles

are so constructed that they "choke up" very easily at the base of the graduated capillary. A slight constriction at this opening will result in preventing some of the fat from rising into the neck of the bottle. By carefully selecting the test bottles and eliminating those that repeatedly "choked up" this difficulty was lessened. For this reason, in selecting bottles that are to be used in the test, care should be taken to choose those that have the graduations close together, as this indicates a wide capillary. Too much emphasis cannot be laid upon the need of a test bottle that is properly constructed for the testing of buttermilk.

The normal butyl alcohol test also is somewhat handicapped by the lack of a desirable test bottle; otherwise the method is very successful.

CONCLUSIONS

1. The amount of acid used has a very important effect upon the results obtained in the Babcock method of testing buttermilk.

2. The extension of the whirling period beyond the 10-10-5 minute periods has no appreciable effect upon the result obtained in the Babcock method of testing buttermilk.

3. Increasing the speed of the centrifuge above 900 to 1000 revolutions per minute will not increase the readings obtained in the Babcock method of testing buttermilk.

4. With the proper modifications of the Babcock test and with the addition of a correction factor of 0.2, results can be obtained that will conform closely to the Roese-Gottlieb determination.

5. The normal butyl alcohol method of testing buttermilk for fat checks closely with the Roese-Gottlieb method.

DIRECTIONS FOR OPERATING THE MODIFIED BABCOCK TEST FOR BUTTERMILK

As a result of this investigation, the following modification of the Babcock test for determining the fat in buttermilk is presented:

1. If the sample contains visible granules of butter fat, strain it thru cheesecloth, as floating granules of butter are not indicative of the exhaustiveness of churning.

2. Thoroly mix the sample.

3. By means of a 10-gram Mojonnier pipette, measure 10 grams of the buttermilk to be tested into a skim-milk bottle (use a 0.5-percent bottle if possible).

4. Add 13.5 cc. of commercial sulfuric acid (having a specific gravity of 1.82–1.83) from a burette or 14 cc. from an acid measure, and mix well.

5. Centrifuge at a speed of about 1000 R.P.M. for 10 minutes.

6. Add enough hot soft water to raise the mixture in the bottle to the point where the graduated neck is joined to the shoulder of the bottle. Whirl 10 minutes.

7. Finish filling bottle with hot soft water and whirl for 5 minutes.

8. Allow to remain 5 minutes in a hot-water bath having a temperature of 135–140° F.

9. Read to top of meniscus and multiply the result by 1.8. (The fat column should be a dark amber color. If it is a straw yellow color, the amount of acid used should be increased.)

10. Add the correction factor of 0.2.